P32853

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Bernhard H. VAN LENGERICH T.C. Art Unit: 1612

Serial No.: 09/782,320 Examiner: ROBERTS, Lezah W.

Filed : February 13, 2001 Confirmation No.: 9819

For: **EMBEDDING AND ENCAPSULATION OF SENSITIVE COMPONENTS INTO A MATRIX TO OBTAIN DISCRETE CONTROLLED RELEASE PARTICLES**

REQUEST FOR REFUND

United States Patent and Trademark Office
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Sir:

Applicant respectfully requests a refund in the amount of $500.00 that was charged to our Deposit Account No. 19-0089 on July 18, 2011 in connection with the filing of the Appeal Brief filed on July 1, 2011.

The basis of this Request for Refund is that our Deposit Account was charged $540.00 for the fees of filing a brief in support of an appeal. However, the Appeal Brief filed on July 1, 2011 is the second Appeal filed in this case, with the first Appeal Brief being filed via certificate of facsimile transmission on January 12, 2007 with the then requisite fee of $500.00. All rejections were withdrawn in a September 4, 2008 Office Action reopening prosecution. A final Board decision was not rendered on that previous Appeal. Accordingly, pursuant to MPEP 1204.01 the $500.00 previously paid Appeal fee should be applied to the present Appeal, and so only the $40.00 increase from the previously paid fee of $500.00 to the current requisite $540.00 fee for filing an Appeal Brief under 37 C.F.R. § 41.20(b)(2) should have been charged.

Copies of the Appeal Brief filed on January 12, 2007 and the fee transmittal receipt for deducting the appeal fee in the amount of $500.00 on March 9, 2007 indicating that the payment was authorized and was received are enclosed herewith.

Please make payment of the refund in the amount of $500.00 to Deposit Account No. 19-0089.

Respectfully submitted,
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CERTIFICATE OF FACSIMILE TRANSMISSION

This is to certify that this document entitled APPEAL BRIEF is being faxed to the Commissioner for Patents / Examiner Michel GRAFFEO at fax no. 703-872-9306 this 12th day of January, 2007.

Phyllis O Caldwell

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.	:	09/782,320 Confirmation No. 9819
Applicant	:	Bernhard H. van LENGERICH
Filed	:	02/13/2001
TC/A.U.	:	1614
Examiner	:	GRAFFEO, Michel
Docket No.	:	BVL-102A
Customer No.	:	23290

APPEAL BRIEF

Mail Stop Appeal Brief-Patents
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

Applicants respectfully submit the following Appeal Brief in accordance with 37 C.F.R. 41.37 together with the fee set forth in 37 CFR 41.20(b).

This Appeal is from a final Office Action dated June 15, 2006, finally rejecting Claims 25, 27-31, 34, 35, 37-40, 42, 46, 52-59, 61-62, 64-67, 69, 70, 73, 75, 79, 81-85, 91-93, 95-97, 101, 103 and 105. No claims are allowed.

I. REAL PARTY IN INTEREST

The real party in interest is General Mills, Inc., by way of Assignments recorded in the U.S. Patent and Trademark Office at Reel 011062, Frame 0728, and Reel 011062, Frame 0788, on October 25, 2000.

II. RELATED APPEALS AND INTERFERENCES

There are presently no related appeals or interferences known to Applicants, Applicants' representatives, or the Assignee.

III. STATUS OF CLAIMS

Claims 1-24, 32-33, 36, 41, 43-45, 47-49, 51, 60, 63, 68, 71-72, 74, 76-78, 80, 86-90, 98-100, 102, 104, and 106-107 have been canceled.

Claims 25, 27-31, 34-35, 37-40, 42, 46, 52-59, 61, 62, 64-67, 69-70, 73, 75, 79, 81-85, 91-93, 95-97, 101, 103, and 105 are pending and stand rejected. Claims 26, 50, and 94 are withdrawn from consideration. Pending Claims 25, 27-31, 34-35, 37-40, 42, 46, 52-59, 61, 62, 64-67, 69-70, 73, 75, 79, 81-85, 91-93, 95-97, 101, 103, and 105 are being appealed.

In the September 16, 2004 Ex Parte Quayle Action, the elected species indicated as allowable were: (1) durum wheat as the plasticized matrix material and (2) a probiotic neutraceutical component as an encapsulant. Applicant notes that the election of species requirement directed to the rate-controlling agent (hydrophobic component/fat) was withdrawn in the Office Action dated March 17, 2003. As to the additional matrix material, Applicant elected starch as the additional matrix material in Claim 79. As to the encapsulant form, Applicant elected the liquid encapsulant of Claim 93. In the December 13, 2005 the Examiner chose plasticized starch as the next species for the plasticized matrix material for examination.

The elected species of durum wheat as a plasticized matrix material was therefore indicated as allowable because the Examiner chose plasticized starch as the next species of plasticized matrix material to be examined. See the paragraph bridging pages 4 and 5 of the June 16, 2006 Final Rejection. Thus, with respect to the originally elected species of durum wheat as a plasticized matrix material, claims readable thereon and which should be indicated as allowable with respect to durum wheat as the plasticized matrix material are Claims 25, 27-31, 34-35, 37-40, 42, 46, 52, 54-59, 61-62, 64-67, 69-70, 73, 75, 79, 81-85, 91-93, 95-97, 101, 103, and 105. Also, Applicant respectfully asserts that withdrawn Claims 26 and 50, like examined, pending Claim 53, recite plasticized starch as a matrix material and thus should not be indicated as withdrawn, but as pending in view of the Examiner's selection of plasticized starch as the next species for examination.. The claims are set forth in the attached Claims Appendix.

IV. STATUS OF AMENDMENTS

An Amendment After Final Rejection Under 37 C.F.R. 1.116 and Summary of Personal Interview, and an accompanying Supplemental Information Disclosure Statement were filed via certificate of mailing dated September 15, 2006. A Notice of Appeal was filed on November 15, 2006. An Advisory Action was mailed November 16, 2006, indicating entry of the September 15, 2006 Amendment After Final Rejection Under 37 C.F.R. 1.116 and Summary of Personal Interview, and entry of the accompanying Supplemental Information Disclosure Statement with an initialed Form PTO-1449.

V. SUMMARY OF CLAIMED SUBJECT MATTER

Independent claim 26 is directed to an encapsulated product comprising discrete, solid particles having a substantially uniform shape and a diameter of up to about 10 mm. See page 7 lines 4-6 and17-20, page 9 line 26 to page 10 line 1, and page 28 line 30 to page

29 line 8 and FIGS. 4 and 5. Each particle comprises an encapsulant dispersed throughout a plasticized mass, and at least one component for controlling the rate of release of the encapsulant. The plasticized mass comprises at least about 40% by weight of at least one matrix material, based on the weight of the final product, and at least one plasticizer. The encapsulant and plasticized matrix material form an at least substantially homogeneous mixture. See page 6 lines 2-4, page 7 lines 4-12, page 8 lines 2-11, page 8 line 22 to page 9 line 15, page 10 lines 3-11, page 11 lines 2-16, page 11 line 27 to page 14 line 3, page 22 line 12 to page 23 line 23, and page 26 lines 17-19. The encapsulant is at least one pharmaceutical component, neutraceutical component, nutritional component, fragrance component, or biologically active component in an amount of from about 1% by weight to about 85% by weight, based upon the weight of the matrix material. See page 10 lines 11-15, page 11 lines 19-23, page 14 line 9 to page 10 line 2, and page 21 lines 11-19.

Independent claim 52 is also directed to an encapsulated product comprising discrete, solid particles having a substantially uniform shape. See page 7 lines 4-6 and17-20, page 9 line 26 to page 10 line 1, and page 28 line 30 to page 29 line 8 and FIGS. 4 and 5. Each particle comprises an encapsulant dispersed throughout a plasticized matrix material, and at least one component for controlling the rate of release of the encapsulant. The plasticized matrix material comprises at least one member selected from the group consisting of durum wheat, semolina, wheat flour, wheat gluten, soy protein, hydrocolloids, casein, and gelatin, and at least one plasticizer. The amount of the plasticized matrix material is at least about 40% by weight, based on the weight of the final product. The encapsulant and plasticized matrix material form an at least substantially homogeneous mixture. See page 6 lines 2-4, page 7 lines 4-12, page 8 lines 2-11, page 8 line 22 to page 9 line 15, page 10 lines 3-11, page 11 lines 2-16, page 11 line 27 to page 14 line 3, page 22 line 12 to page 23 line 23, page 26 lines 17-19, page 33 lines 17-20, page 42 Table 2, and FIG. 3. The encapsulant is at least one pharmaceutical component, neutraceutical component, nutritional component,

fragrance component, or biologically active component in an amount of from about 1% by weight to about 85% by weight, based upon the weight of the matrix material. See page 10 lines 11-15, page 11 lines 19-23, page 14 line 9 to page 10 line 2, and page 21 lines 11-19.

Independent claim 83 is directed to the same product as independent claim 52 but does not recite wheat flour in the Markush grouping of plasticized matrix materials. Also, claim 83 explicitly recites that the amount of encapsulant is from about 3% by weight to about 50% by weight, based upon the weight of the matrix material. See page 21 lines 15-19.

VI. GROUNDS OF REJECTION TO BE REVIEWED UPON APPEAL

1. Claims 25, 27-31, 34, 35, 37-40, 42, 46, 52-59, 61-62, 64-67, 69, 70, 73, 75, 79, 81-85, 91-93, 95-97, 101, 103 and 105 stand rejected under 35 U.S.C. 103(a) as obvious over U.S. Patent No. 4,187,321 (Mutai et al.).

VII. ARGUMENT

A. Mutai et Al Does Not Teach or Suggest a Plasticized Mass or Plasticized Matrix Material as Recited in Independent Claims 25, 52, and 83 and Their Dependent Claims

Mutai et al. discloses foods and drinks containing bifidobacteria prepared by growing, in a milk medium under aerobic conditions, a mixture of bifidobacteria containing a mutant strain of oxygen-resistant Bifidobacterium and a strain of anaerobic Bifidobacterium (Abstract). The Examiner cites Example 3 for disclosing starch and col. 2, lines 35-43 for disclosing fat. Example 3 discloses aerobically culturing three species of bacteria in a semi-synthetic medium at a temperature of 37°C for 20 hours. The culture was introduced into a freeze centrifuge for separation and collection of cells from the culture. The freeze dried cells obtained from the culture were dispersed in a medium containing skim

milk (10%), sodium glutamate (1.0%), and Vitamin C (1%) and were freeze-dried to obtain a dried material containing three species of bacteria. The resultant freeze dried material was mixed with a 20-fold volume of dried starch and tableted. Col. 2, lines 35-43 discloses that the media for cultivating the bacteria may be whole milk.

Mutai et al. does not teach or suggest an encapsulated product comprising discrete, solid particles wherein each particle comprises an encapsulant dispersed throughout a plasticized mass or plasticized matrix material, as recited in independent Claims 25, 52, and 83 and their dependent claims. The dried starch of Mutai et al is simply not a plasticized matrix material and no evidence has been presented to indicate that dried starch is plasticized as alleged by the Examiner.

Furthermore, the two ingredients which are mixed together and tableted (the dried starch and the freeze dried material containing the cells) are not combined with a plasticizer to plasticize dried starch. The dried ingredients are merely powders which are pressed together to form a tablet with no plasticization of the dried starch. The freeze dried material containing the cells, even though it was obtained from cells and skim milk, does not inherently function as a plasticizer to plasticize the dried starch. The liquid from the cell culture medium and the skim milk is removed by freeze drying and is unavailable to plasticize the 20-fold volume of dried starch. Also, the temperature employed during mixing of the dried starch and the dried material containing the cells is too low to gelatinize or melt the dried starch so as to plasticize it.

As discussed in the paragraph bridging pages 8-9 of the specification, at least one plasticizable, matrix-forming material such as starch may be admixed with a sufficient amount of a plasticizer such as water to reduce the melt or glass transition temperature of the plasticizable material, together with the additional release-rate controlling ingredient. The mix is heated above the melt or glass transition temperature of the plastifiable or matrix material, such as above the gelatinization temperature of a starch matrix ingredient, while

conveying and mixing the ingredients within an extruder. The temperature is maintained sufficiently high for a sufficiently long period of time to at least partially gelatinize starch in the mixture.

As disclosed in the paragraph bridging pages 22 and 23 of the specification, in embodiments where starch is used as a matrix material, the starch is at least partially gelatinized without substantially destructurizing and dextrinizing the starch. The degree of gelatinization may, for example, be at least about 75%, for example, at least about 90%, or essentially completely gelatinized. In embodiments of the invention, to achieve at least substantial gelatinization of starch, the starch and plasticizer (preferably water) admixture may be maintained at a temperature of the blend of at least about 100°C, preferably from about 120°C to about 150°C, for example, from about 125°C to about 140°C, for a period of time of at least about 3 l/d preferably about 5 to 7 l/d of extruder length. For example, for starches having an amylose content of more than about 25%, for example about 50% to about 70%, it may be necessary to maintain a product temperature inside the extruder of about 125°C for a sufficient amount of time, for example for about 4 l/d, preferably about 7 to 8 l/d of extruder length at a low screw rotational rate of about 150 to about 200 rpm using medium pitch screw elements to assure at least substantial gelatinization of the starch. These temperatures are much higher than the 37°C culturing temperature employed by Mutai et al and would destroy the cultured cells if used to plasticize Mutai et al's mixture of dried starch and dried material containing the cells. Use of processing conditions which would destroy the cultured cells is contrary to the disclosure of Mutai et al. See col. 3. lines 5-7.

There is no teaching or suggestion that the "dried starch" of Example 3 of Mutai et al. is a plasticized material and dried starch is not inherently plasticized. Contrary to the Examiner's assertion, the products as claimed which contain a plasticized starch matrix material in an amount of at least about 40% by weight, based upon the weight of the final

product, are not the same as that in the art and having substantially different, controlled release properties.

Starch production does not inherently result in a plasticized starch even if heat treatment is utilized in such processes. Any heat treatment up to about 50°C that occurs during starch processing does not result in plasticization, as shown by the references submitted with applicant's September 15, 2006 Information Disclosure Statement.

Pomeranz, *Wheat is Unique*, discloses that the processing of wheat according to a modified Longford-Slotter process occurs with unmilled wheat being steeped in the laboratory for 15 hours at 37°C (page 521). See also pages 524-525 (Alsatin process) and 528 (Halle process). Similarly, in discussing the wet milling of wheat flour, Cornell et al. in *Wheat Chemistry and Utilization* states that at temperatures below 35°C, the starch does not gel and is able to be removed by centrifugation (pages 79-80). Likewise, in White et al., *Corn: Chemistry and Technology*, page 465 it states that the water used for corn starch washing:

> is heated to 38-43°C to enable removal of soluble matter ... Temperature sensors in the hydroclone systems protect the starch slurry from temperature exceeding 54°C, well below the starch-pasting temperature of 63°C. (Emphasis added).

In contrast, Whistler et al., *Starch: Chemistry and Technology*, show that the pasting or gelatinization temperature of starch is in the range of 52-63°C for wheat and 62-72°C for corn (Table 1, page 292). Likewise Table 6.1 on page 121 of Whistler et al., *Carbohydrate Chemistry for Food Scientists*, indicates gelatinization/pasting temperatures are in the range of 52-85°C for wheat and 62-80°C for corn.

Thus, starch production, particularly starch extraction, does not inherently result in plasticization of the starch because it occurs at temperatures lower than the pasting or

gelatinization temperature. Further, plasticization/gelatinization causes disruption of the molecular order within starch granules, as evidenced by irreversible granule swelling, loss of birefringence, and loss of crystallinity (page 128 of Whistler et al., *Carbohydrate Chemistry for Food Scientists*).

In addition, Applicant demonstrated to the Examiner that non-plasticized starch or native wheat starch, such as the dried starch of Mutai et al., is structurally different than plasticized starch and therefore has substantially different release properties from those of plasticized starch. As set forth in the September 15, 2006 Amendment After Final Rejection Under 37 C.F.R. 1.116 and Summary of Personal Interview, at the August 17, 2006 personal interview, Applicant's representative placed 0.5 mm pellets comprising plasticized hard wheat flour in water in a first clear plastic cup and placed 0.5 mm pellets comprising non-plasticized native wheat starch in water in a second plastic cup. All pellets were made by adding water to flour or starch inside an extruder, extruding the mixture through a die, cutting the extrudate into pieces, and then drying the pieces at room temperature.

After intermittent stirring of both plastic cups, the pellets comprising the native wheat starch quickly disintegrated and clouded the water. In contrast, the pellets comprising the plasticized hard wheat flour remained intact, although there was very slight cloudiness due to residual starch granules that are so small that they remained in suspension and settled over time.

In the November 16, 2006 Advisory Action, the Examiner alleges that notwithstanding the above documents submitted by Applicants, "plasticized can be interpreted to mean deformable and to that extent it is taught in Mutai." Mutai does not mention that the dried starch is deformable and no evidence or reference has been cited to support the interpretation that plasticized also means deformable. In addition, the tenses of the terms used by the Examiner are different. Plasticized is a past tense and means that plasticization has taken place. However, deformable means deformation is possible and

therefor plasticization is possible. It is not seen how the dried starch has been deformed or plasticized. The Examiner is confusing "plasticizable" with "plasticized." Dried starch is plasticizable upon the addition of a plasticizer, such as water, and the application of heat, as explained in detail above and in the specification. However, Mutai et al do not add a plasticizer such as water to the dried starch and do not heat the dried starch so as to obtain a plasticized mass or plasticized matrix material as claimed.

In the November 16, 2006 Advisory Action, the Examiner also states that "the instant Specification recites possible starches and sources thereof on page 12 which simply include flours from grains such as corn, wheat, durum wheat etc. and does not require a process for the plasticization of the claimed mass." However, the starches and sources thereof which are recited are examples of plasticizable matrix materials, not plasticized matrix materials, as evidenced by use of the phrase "The matrix material may be a plasticizable biopolymer such as.." at page 11 line 27. A process for the plasticization of the plasticizable starches and sources thereof to obtain the claimed plasticized mass or claimed plasticized matrix material involves heating and the addition of a plasticizer such as water as described above and in the present specification at inter alia page 7 lines 7-15, page 8 line 22 to page 9 line 15, page 22 line 12 to page 23 line 14, page 33 line 17 to page 34 line 9, and FIG. 3.

B. Mutai et al Does Not Teach or Suggest the Controlled Release Properties of Claims 38 and 65

Mutai et al does not teach or suggest an encapsulated product where the encapsulant is released in an aqueous or gastric juice environment in an amount of no more than from about 10% in about 1 hour to no less than about 90% in about 24 hours as claimed in Claims 38 and 65. As discussed above and as set forth in the September 15, 2006 Amendment After Final Rejection Under 37 C.F.R. 1.116 and Summary of Personal Interview, and as

demonstrated at the August 17, 2006 personal interview, non-plasticized starch or native wheat starch, such as the dried starch of Mutai et al., is structurally different than plasticized starch and therefore has substantially different release properties from those of plasticized starch. The pellets comprising the native wheat starch quickly disintegrated and clouded the water. In contrast, the pellets comprising the plasticized hard wheat flour remained intact, although there was very slight cloudiness due to residual starch granules that are so small that they remained in suspension and settled over time.

C. Mutai et al Does Not Teach or Suggest the Amount of Release Agent Claimed in Claims 39 and 81

Mutai et al does not teach or suggest an encapsulated product where the amount of the at least one component used to control the rate of release of the encapsulant is from about 5% by weight to about 50% by weight, based upon the weight of the matrix material as claimed in claims 39 and 81. Even assuming that the fat of the milk used in Example 3 of Mutai et al controls the rate of release of the three species of bacteria, the amount of fat is substantially lower than the amount claimed by applicant in claims 39 and 81 and there is no motivation to increase its level. Mutai et al is not concerned with controlled release of encapsulants.

As discussed above, in Example 3 of Mutai et al, the freeze dried cells obtained from the culture were dispersed in a medium containing skim milk (10%), sodium glutamate (1.0%), and Vitamin C (1%) and were freeze-dried to obtain a dried material containing three species of bacteria. The resultant freeze dried material was mixed with a 20-fold volume of dried starch and tableted. If the dried starch is the matrix material, then the resultant freeze dried material is about 5% by weight, based upon the weight of the starch. The milk was only 10% of the medium which was freeze dried, and the milk fat is only a

few percent of milk, and therefore the fat content of the tablet is substantially less than 5% by weight, based upon the weight of the dried starch.

D. Mutai et al Does Not Teach or Suggest the Plasticized Matrix Materials of Claims 42, 52-59, 61, 62, 64-67, 69-70, 73, 75, 79, 81-85, 95-97, 103, and 105

Mutai et al does not teach or suggest an encapsulated product where the plasticized matrix material comprises at least one member selected from the group consisting of durum wheat, semolina, wheat flour, wheat gluten, soy protein, hydrocolloids, casein, and gelatin. In the paragraph bridging pages 4 and 5 of the June 16, 2006 Final Rejection, the elected species of durum wheat as a plasticized matrix material was indicated as allowable and the Examiner chose plasticized starch as the next species of plasticized matrix material to be examined. However, Claims 42, 52-59, 61, 62, 64-67, 69-70, 73, 75, 79, 81-85, 95-97, 103, and 105 recite plasticized matrix materials other than plasticized starch, none of which are disclosed or suggested by Mutai et al. These plasticized matrix materials have different compositions, such as proteins, and different release properties from those of the dried starch of Mutai et al as demonstrated in Examples 4 to 8 and Comparative Example 2 of the present specification. Mutai et al is not concerned with controlled release of encapsulants and there is no motivation to include any of the specifically claimed plasticized matrix materials in the compositions of Mutai et al.

VIII. CONCLUSION

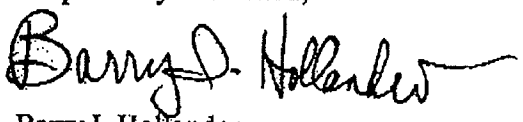
For all of the above reasons, Applicants respectfully request this Honorable Board to reverse the rejection of Claims 25, 27-31, 34-35, 37-40, 42, 46, 52-59, 61, 62, 64-67, 69-70, 73, 75, 79, 81-85, 91-93, 95-97, 101, 103, and 105.

The Commissioner is hereby authorized to charge the appeal fee in the amount of $500 to Deposit Account No. 501032. Any additional fees should be charged to, or any

Serial No. 09/782,320
APPEAL BRIEF
Attorney Docket No. BVL-102A

The Commissioner is hereby authorized to charge the appeal fee in the amount of $500 to Deposit Account No. 501032. Any additional fees should be charged to, or any overpayment in fees should be credited to, Deposit Account No. 501032 (Docket No. BVL-102A).

Respectfully submitted,



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Date: January 12, 2007

CLAIMS APPENDIX

1-24. (Canceled)

25. (Previously presented) An encapsulated product comprising discrete, solid particles having a substantially uniform shape and a diameter of up to about 10 mm, wherein each particle comprises:

an encapsulant dispersed throughout a plasticized mass, and at least one component for controlling the rate of release of the encapsulant,

wherein said encapsulant is at least one pharmaceutical component, neutraceutical component, nutritional component, fragrance component, or biologically active component,

wherein said plasticized mass comprises at least about 40% by weight of at least one matrix material, based on the weight of the final product, and at least one plasticizer,

wherein the encapsulant and plasticized matrix material form an at least substantially homogeneous mixture, and

wherein the amount of said encapsulant is from about 1% by weight to about 85% by weight, based upon the weight of the matrix material.

26. (Withdrawn) An encapsulated product according to claim 25 wherein said plasticized matrix material comprises an at least partially gelatinized starch, which starch is not substantially dextrinized.

27. (Original) An encapsulated product according to claim 25 wherein said encapsulant is coated with a film-forming material prior to dispersion within said plasticized mass.

28. (Original) An encapsulated product according to claim 25 wherein said particles are in the form of a tablet, or a pellet.

29. (Original) An encapsulated product according to claim 28 wherein said particles are coated with a film-forming material.

30. (Original) An encapsulated product according to claim 25 wherein said at least one release-rate controlling component is a hydrophobic component.

31. (Original) An encapsulated product according to claim 30 wherein said hydrophobic component is at least one member selected from the group consisting of fats, oils, waxes, fatty acids, emulsifiers, polyolefins, paraffin, polyvinyl acetate and derivatives thereof, and modified starches.

32-33. (Canceled)

34. (Original) An encapsulated product according to claim 25 which has a specific density of from about 800 g/liter to about 1500 g/liter.

35. (Previously presented) An encapsulated product according to claim 25 wherein the length-to-diameter ratio of said particles is from about 0.1 to about 10.

36. (Canceled)

37. (Original) An encapsulated product according to claim 25 wherein said particles have a substantially non-expanded, substantially non-cellular structure.

38. (Original) An encapsulated product according to claim 25 wherein said encapsulant is released in an aqueous or gastric juice environment in an amount of no more than from about 10% in about 1 hour to no less than about 90% in about 24 hours.

39. (Previously presented) An encapsulated product according to claim 25 wherein:

the amount of the matrix material is from about 60% by weight to about 95% by weight, based upon the weight of the final product, and

the amount of said at least one component used to control the rate of release of the encapsulant is from about 5% by weight to about 50% by weight, based upon the weight of the matrix material.

40. (Original) An encapsulated product according to claim 39 wherein said particles have a diameter of from about 0.5 mm to about 5 mm and a length-to-diameter ratio of about 0.5 to about 2.

41. (Canceled)

42. (Original) An encapsulated product according to claim 25 wherein said plasticized matrix comprises durum wheat or semolina.

43-45. (Canceled)

46. (Previously presented) An encapsulated product according to claim 25 wherein said encapsulant is at least one member selected from the group consisting of antioxidants, phytochemicals, hormones, microorganisms, prebiotics, probiotics, enzymes, formulations containing zidovudine, macromolecular polypeptides, aromatic nitro and

nitroso compounds and their metabolites useful as anti-viral and anti-tumor agents, HIV protease inhibitors, antibiotics, viruses, steroids, oligopeptides, dipeptides, amino acids, fragrance components, adenosine derivatives, sulfated tannins, monoclonal antibodies, and metal complexes of water-soluble texathyrin.

47-49. (Canceled)

50. (Withdrawn) An encapsulated product according to claim 25 wherein said plasticized matrix material is at least one member selected from the group consisting of starches, cyclodextrins, dextrins, monosaccharides, disaccharides, polyvinylpyrrolidone, copolymers of N-vinylpyrrolidone and vinyl acetate, polyvinyl alcohol, cellulose esters, cellulose ethers, and polyethylene glycol.

51. (Canceled)

52. (Previously presented) An encapsulated product comprising:

discrete, solid particles having a substantially uniform shape wherein each particle comprises:

a plasticized matrix material in an amount of at least about 40% by weight, based on the weight of the final encapsulated product,

an encapsulant dispersed throughout the plasticized matrix material, and

at least one component for controlling the rate of release of the encapsulant,

wherein said encapsulant comprises at least one pharmaceutical component, neutraceutical component, nutritional component, fragrance component, or biologically active component,

wherein said matrix material comprises at least one member selected from the group consisting of durum wheat, semolina, wheat flour, wheat gluten, soy protein, hydrocolloids, casein, and gelatin, and at least one plasticizer,

wherein the encapsulant and plasticized matrix material form an at least substantially homogeneous mixture, and

wherein the amount of said encapsulant is from about 1% by weight to about 85% by weight, based upon the weight of the matrix material.

53. (Original) An encapsulated product according to claim 52 wherein said plasticized matrix material comprises an at least partially gelatinized starch, which starch is not substantially dextrinized.

54. (Original) An encapsulated product according to claim 52 wherein said encapsulant is coated with a film-forming material prior to dispersion within said plasticized mass.

55. (Original) An encapsulated product according to claim 52 wherein said particles are in the form of a tablet, or a pellet.

56. (Original) An encapsulated product according to claim 52 wherein said particles are spherical.

57. (Original) An encapsulated product according to claim 55 wherein said particles are coated with a film-forming material.

58. (Original) An encapsulated product according to claim 52 wherein said at least one release-rate controlling component is a hydrophobic component.

59. (Original) An encapsulated product according to claim 58 wherein said hydrophobic component is at least one member selected from the group consisting of fats, oils, waxes, fatty acids, emulsifiers, polyolefins, paraffin, polyvinyl acetate and derivatives thereof, and modified starches.

60. (Canceled)

61. (Original) An encapsulated product according to claim 52 which has a specific density of from about 800 g/liter to about 1500 g/liter.

62. (Previously presented) An encapsulated product according to claim 52 wherein the length-to-diameter of said particles is from about 0.1 to about 10.

63. (Canceled)

64. (Original) An encapsulated product according to claim 52 wherein said particles have a substantially non-expanded, substantially non-cellular structure.

65. (Original) An encapsulated product according to claim 52 wherein said encapsulant is released in an aqueous or gastric juice environment in an amount of no more than from about 10% in about 1 hour to no less than about 90% in about 24 hours.

66. (Original) An encapsulated product according to claim 52 wherein the amount of said at least one component for controlling the rate of release of the encapsulant is up to about 70% by weight, based on the weight of the matrix material.

67. (Original) An encapsulated product according to claim 52 wherein said particles have a diameter of from about 0.5 mm to about 5 mm and a length-to-diameter ratio of about 0.5 to about 2.

68. (Canceled)

69. (Original) An encapsulated product according to claim 52 wherein said matrix material comprises at least one member selected from the group consisting of durum wheat, semolina, wheat flour, wheat gluten, and soy protein.

70. (Original) An encapsulated product according to claim 52 wherein said matrix material comprises at least one member selected from the group consisting of durum wheat and semolina.

71-72. (Canceled)

73. (Original) An encapsulated product according to claim 52 wherein said discrete, solid particles have a diameter of up to about 10 mm.

74. (Canceled)

75. (Previously presented) An encapsulated product according to claim 52 wherein said encapsulant is at least one member selected from the group consisting of antioxidants, phytochemicals, hormones, microorganisms, prebiotics, probiotics, enzymes, formulations containing zidovudine, macromolecular polypeptides, aromatic nitro and nitroso compounds and their metabolites useful as anti-viral and anti-tumor agents, HIV protease inhibitors, antibiotics, viruses, steroids, oligopeptides, dipeptides, amino acids, fragrance components, adenosine derivatives, sulfated tannins, monoclonal antibodies, and metal complexes of water-soluble texathyrin.

76-78. (Canceled)

79. (Previously presented) An encapsulated product according to claim 52 wherein said plasticized matrix material further comprises at least one member selected from the group consisting of starches, cyclodextrins, dextrins, monosaccharides, disaccharides, polyvinylpyrrolidone, copolymers of N-vinylpyrrolidone and vinyl acetate, polyvinyl alcohol, cellulose esters, cellulose ethers, and polyethylene glycol.

80. (Canceled)

81. (Previously presented) An encapsulated product according to claim 52 wherein:

the amount of the matrix material is from about 60% by weight to about 95% by weight, based upon the weight of the final product, and

the amount of said at least one component used to control the rate of release of the encapsulant is from about 5% by weight to about 50% by weight, based upon the weight of the matrix material.

82. (Original) An encapsulated product according to claim 52 wherein said encapsulant comprises at least one member selected from the group consisting of enzymes and microorganisms.

83. (Previously presented) An encapsulated product comprising discrete, solid particles having a substantially uniform shape wherein each particle comprises:

an encapsulant dispersed throughout a plasticized matrix material, said matrix material comprising at least one member selected from the group consisting of durum wheat, semolina, vital wheat gluten, soy protein, hydrocolloids, casein, and gelatin, and at least one plasticizer,

wherein said encapsulant comprises at least one pharmaceutical component, neutraceutical component, nutritional component, fragrance component, or biologically active component,

wherein the encapsulant and plasticized matrix material form an at least substantially homogeneous mixture,

wherein the amount of said encapsulant is from about 3% by weight to about 50% by weight, based upon the weight of the matrix material, and

wherein the amount of said matrix material is at least about 40% by weight, based upon the weight of the final encapsulated product.

84. (Original) An encapsulated product according to claim 83 wherein said matrix material comprises semolina or durum wheat.

85. (Original) An encapsulated product according to claim 83 wherein said encapsulant comprises at least one member selected from the group consisting of enzymes and microorganisms.

86-90. (Canceled)

91. (Previously presented) An encapsulated product according to claim 25, comprising about 3% by weight to about 50% by weight of the encapsulant, based upon the weight of the matrix material.

92. (Previously presented) An encapsulated product according to claim 25, comprising about 5% by weight to about 20% by weight of the encapsulant, based upon the weight of the matrix material.

93. (Previously presented) An encapsulated product according to claim 25, wherein the encapsulant is in liquid form.

94. (Withdrawn) An encapsulated product according to claim 25, wherein the encapsulant is in solid form.

95. (Previously presented) An encapsulated product according to claim 52, comprising about 3% by weight to about 50% by weight of the encapsulant, based upon the weight of the matrix material.

96. (Previously presented) An encapsulated product according to claim 52, comprising about 5% by weight to about 20% by weight of the encapsulant, based upon the weight of the matrix material.

97. (Previously presented) An encapsulated product according to claim 83, comprising about 5% by weight to about 20% by weight of the encapsulant, based upon the weight of the matrix material.

98-100. (Canceled)

101. (Previously presented) An encapsulated product according to Claim 25 wherein said plasticized mass comprises from about 60% by weight to about 95% by weight of at least one matrix material.

102. (Canceled)

103. (Previously presented) An encapsulated product according to Claim 52 comprising from about 60% by weight to about 95% by weight of the matrix material, based upon the weight of the final encapsulated product.

104. (Canceled)

105. (Previously presented) An encapsulated product according to Claim 83 comprising from about 60% by weight to about 95% by weight of the matrix material, based upon the weight of the final encapsulated product.

106-107. (Canceled)

EVIDENCE APPENDIX

A. Demonstration at Personal Interview

A demonstration regarding functionality of plasticized starch was conducted during an August 17, 2006 Personal Interview as indicated in the August 17, 2006 Interview Summary. The demonstration was described at page 16 of An Amendment After Final Rejection Under 37 C.F.R. 1.116 and Summary of Personal Interview, filed via certificate of mailing dated September 15, 2006. An Advisory Action was mailed November 16, 2006, indicating entry of the September 15, 2006 Amendment After Final Rejection Under 37 C.F.R. 1.116 and Summary of Personal Interview. A copy of page16 from the September 15, 2006 Amendment After Final Rejection Under 37 C.F.R. 1.116 and Summary of Personal Interview is attached.

B. Literature References Relied Upon By Applicants

A Supplemental Information Disclosure Statement was filed via certificate of mailing dated September 15, 2006. An Advisory Action was mailed November 16, 2006, indicating entry of the Supplemental Information Disclosure Statement (SIDS) with an initialed Form PTO-1449. The portions of the references relied upon by Applicant in the Brief which are cited in the SIDS and copies of which are attached are:

1. Pomeranz, *Wheat is Unique*, page 521, pages 524-525 (Alsatin process) and page 528 (Halle process).

2. Cornell et al. in *Wheat Chemistry and Utilization*, pages 79-80.

3. White et al., *Corn: Chemistry and Technology*, page 465.

4. Leach, Whistler et al., *Starch: Chemistry and Technology*, Table 1, page 292.

5. Whistler et al., *Carbohydrate Chemistry for Food Scientists*, Table 6.1 on page 121, and page 128.

of species requirement directed to the rate-controlling agent (hydrophobic component/fat) was withdrawn in the Office Action dated March 17, 2003.

The Examiner chose plasticized starch as the next species of matrix material to be examined, after indicating that the durum wheat species is allowable. Applicant respectfully asserts that Claims 26 and 50 recite plasticized starch as a matrix material and thus should not be indicated as withdrawn, but as pending. For the next elected species, Applicant respectfully requests the Examiner to elect the species of plasticized matrix material already recited in independent Claims 52 and 83 and/or in dependent Claims 42, 69-70, and 84.

III. EXAMINER INTERVIEW

At the August 17, 2006 personal interview, Applicant's representative placed 0.5 mm pellets comprising plasticized hard wheat flour in water in a first clear plastic cup and placed 0.5 mm pellets comprising non-plasticized native wheat starch in water in a second plastic cup. All pellets were made by adding water to flour or starch inside an extruder, extruding the mixture through a die, cutting the extrudate into pieces, and then drying the pieces at room temperature.

After intermittent stirring of both plastic cups, the pellets comprising the native wheat starch quickly disintegrated and clouded the water. In contrast, the pellets comprising the plasticized hard wheat flour remained intact, although there was very slight cloudiness due to residual starch granules that are so small that they remained in suspension and settled over time. Thus, Applicant argued that native wheat starch, such as the dried starch of Mutai et al., is structurally different than plasticized starch and therefore has different properties.

Evidence Appendix A:
Page 16 from Amendment After Final Rejection filed 9/15/2006.

WHEAT IS UNIQUE

STRUCTURE, COMPOSITION, PROCESSING, END-USE PROPERTIES, AND PRODUCTS

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Published by the
American Association of Cereal Chemists
St. Paul, Minnesota, USA

Evidence Appendix B-1:
Pomeranz, *Wheat is Unique*, page 521, pages 524-525 (Alsatin process) and page 528 (Halle process).

31

PROCESS FOR THE INDUSTRIAL PRODUCTION OF WHEAT STARCH FROM WHOLE WHEAT

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ABSTRACT

Of a total of fifteen different processes for the industrial production of wheat starch, six are described in the present paper. The fifteen processes can be grouped according to whether the raw material used is whole wheat or wheat flour and whether the separation technique used is mechanical, chemical, or fermentative. They can also be grouped according to whether the gluten obtained as a by-product is vital or devitalized. The classification used in the present case is that based on the raw material used. Six of the fifteen production processes use whole wheat as the raw material. The paper ends with a description of a modified Longford-Slotter process in which unmilled wheat was steeped in the laboratory for 15 hours at 37 °C with the addition of 0.2 % of sulfur dioxide. With this process it was possible to recover 54 % of starch having a protein content of 0.4 %, corresponding to an efficiency of just under 81 %, with a single discontinuous centrifugation and a single tabling operation.

TABLE III
Classification of processes for the industrial production of wheat starch - separation

Mechanical	Chemical	Fermentative
Alsatin	Dimler	Halle
Alsace	Longford-Slotter	
Martin	Phillips-Sallans	
Fesca	Far-Mar-Co	
Batter	Pillsbury	
Alfa-Laval-Raisio		
Verberne-Zwitserloot		
Weipro		
Tenstar		

the one hand and washing out, purifying, and drying the starch on the other. Gluten recovery, in contrast, is only of minor importance. In comparison with processes using wheat flour as the raw material, the use of whole wheat has the important processing advantage that wheat having characteristic physical and chemical properties is always available and ready for use. The possibility of supplyside bottlenecks is avoided, since there is no dependence on the milling industry. Other advantages are the substantially higher yields of first-grade starch, as a result of reduced damage to the starch granules, and an improvement in the vitality of the gluten in some cases, since the separation of the endosperm protein is almost complete.

Alsatin process (Kerr, 1950, Radley, 1953). The Alsatin process, which was developed in the USA, is based on the observation that steeping the wheat in warm water not only makes grinding considerably easier, but also leads to higher starch yields, with a simultaneous improvement in quality (Fig. 1).

The steeping with water is carried out at 30 to

: industrial
:paration

Fermentative

Halle

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TABLE IV
Classification of processes for the industrial production of wheat starch - gluten

Vital	Devitalized
Alsatin	Halle
Martin	Alsace
Fesca	Dimler
Batter	Longford-Slotter
Alfa-Laval-Raisio	Phillips-Sallans
Verberne-Zwitserloot	Pillsbury
Weipro	
Far-Mar-Co	
Tenstar	

TABLE V
Classification of processes for the industrial production of wheat starch - whole wheat as raw material

Alsatin	Longford-Slotter
Halle	Far-Mar-Co
Alsace	Pillsbury

35 °C and takes between 24 and 48 hours. Continuous renewal of the steep water helps to wash out soluble material, and at the same time prevents the acidity from becoming too high. The steeping is followed by coarse grinding between either millstones or fluted rollers. The resulting dough is thoroughly kneaded in extractors consisting of a finely perforated with rotating mixing elements, and the starch/gluten suspension is simultaneously washed with a continuous supply of fresh water from a spray, with separation of

The cleaned wheat is first steeped, an operation that is vitally important to the starch yield, and whose duration depends on the type of wheat; the fermentation may then be carried out either during the steeping process, immediately after grinding, or during both of these stages. Regardless of the variant adopted, the fermentation, which is initiated by the addition of so-called acid water, takes about a week and proceeds at a temperature of about 25 C. Apart from small quantities of alcohol, the early stages of fermentation lead mainly to the formation of organic acids with the aid of acid-forming bacteria. The end of fermentation is marked by the start of putrefaction, which is accompanied by the appearance of a strong, unpleasant odor due to the decomposition of proteins. This odor signals the end of fermentation and also of the desired action on the gluten, which should be sufficiently degraded by the fermentation to make its separation from the starch as easy as possible. When fermentation is complete and the acid water has been removed, the starch is washed out in rotating perforated drums. The resulting starch slurry leaves the drum through openings in the wall, while the gluten and the hull fractions remain inside the drum. The starch slurry can then be purified either by settling and screening in stirred tubs or by centrifugation. The refining and concentration stages are followed by drying. Starch yields of 60 % can be achieved by the Halle process, with low protein contents. An undesirable side effect of this process, however, is the unsatisfactory smell of the starch. The bran is washed, dewatered, and dried and then marketed as animal feed.

Though the Halle process was used for many centuries until the French apothecary Martin developed the production of starch from wheat flour in 1835, it has now been totally replaced by other processes, since the destruction of the gluten meant that the recovery of marketable gluten was impossible. The profitability of a wheat starch factory using the Halle process was thus severely limited by the loss of the gluten, which would have commanded high prices on the market (Parow, 1928, Singer, 1937,Kerr, 1950,

WHEAT
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Note statement on p 79-80

Evidence Appendix B-2:
Cornell et al. in *Wheat Chemistry and Utilization*, pages 79-80.

., and Vandercook, C.E.

Chem. 70:77.

ıbo, T., and Odashima S.

Chem. 29:407.
eal Chem. 67:434.
)1.

Technol. 22(3):76.
al Chem. 66:329.
99.

ling In: Wheat and Wheat
American Society Agron-

al Chem. 34:337.
Cereal Chem. 39:155.

. Chem. 67:421.
/ and Technology, Ed. Y.
St. Paul, Minn.) Chap. 4.

CHAPTER 3

The Wet Milling of Wheat Flour

3.0 INTRODUCTION

THE process for the wet milling of wheat flour is quite different from that for the wet milling of corn, the latter being by far the largest process of this type in the United States, accounting for over 95% of the starch production in that country.

The wet milling of wheat flour has the same purpose as that for corn—the production of starch—but in addition it is able to yield high-quality gluten, suitable for food supplementation. Modern wet-milling processes for corn are applicable to wheat grain, but the quality of the gluten manufactured is not high enough to merit its use, keeping in mind that protein quality is paramount to its use in bakery products. Processing of corn, instead, places great importance on recovery of corn oil.

Wheat starch manufacturers have found it advantageous to use flour rather than whole wheat as the raw material. Wheat flour has a higher starch content than the grain; there are far fewer by-products (bran, fiber, germ), and consequently the recovery of the starch and gluten can be achieved with less plant. However, it has to be recognized that these advantages are partially offset by its higher cost. A high-quality white flour is required for the manufacture of starch and gluten; whole-meal flour is not used.

3.1 TYPES OF PROCESSES

3.1.1 THE ALKALI PROCESS

The earliest process used for wheat flour probably stems from a time-honored process for rice whereby extraction of protein is achieved by means of alkalies. For example, 0.03 mol/L sodium hydroxide (1 L) is able to give a complete protein dispersion when mixed with the flour (150 g) for 1 h when the pH is usually 11.4–11.7. At temperatures below 35°C, the

starch does not gel and is able to be removed by centrifugation. The protein is recoverable in yields of about 75% from the supernatant liquors by reducing the pH to say 5.5 and again centrifuging. When dried, the protein content is about 85% (dry basis, N × 5.7).

The wheat protein so recovered, has been denatured and would not be generally suitable for addition to baked goods. However, it could be utilized as a protein supplement where reconstitution in water without production of the elastic form of the protein (vital gluten) is actually an advantage.

The best quality starch recovered (about 70% of the total starch) has a protein content of 0.3% (dry basis, N × 5.7), while the remainder (20%) normally contains about 0.6% protein.

3.1.2 THE MARTIN PROCESS

The so-called "Martin" process has been employed since the 1920s and is still used today in modified form. It is based on forming a dough, washing the starch out of the dough, and recovering the gluten as a vital (undenatured) product. In a series of experiments, Shewfelt and Adams (1945, 1946) evaluated several flours and techniques. In most cases, flour was doughed with about 85% of its weight of water. Because of the nature of the gluten, most of it is readily recovered as curds upon addition of water, while the remainder can be recovered on vibratory screens.

The starch is recovered from the liquors ("milk") by centrifugal methods. Many of the steps require washing with water in a continuous manner, e.g., by hydrocyclones (Svarovsky 1984) and concentration of the starch liquors before the final step—the dewatering or recovery of the starch "cake" (see Section 3.2).

The gluten produced can be dried to about 90% solids content in order to make it available for distant, e.g., overseas, markets. This can be carried out without significant denaturation of the proteins and the product is marketed as "dry vital gluten," generally of protein content about 75% (83% dry basis, N × 5.7). Recovery of gluten is normally about 93–95%, allowing for starch nitrogen and soluble nitrogen in the flour. Well-washed gluten can be as high as 90% protein on a dry basis. Some of the gluten can be deliberately denatured if it is required for special purposes, e.g., diabetic bread and high-protein food supplements.

The yield of high-grade starch from the Martin process is normally about 60% of the weight of flour (both are normally about 12% moisture content). Up to about 15% of second-grade starch is also produced by concentration of liquors using hydrocyclones. The first-grade starch is superior because of its having:

(a) A higher percentage of large granule starch
(b) Lower lipid content

CORN:
Chemistry and Technology
Second Edition

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Note Statements on pages 465 & 472

Evidence Appendix B-3:
White et al., *Corn: Chemistry and Technology*, page 465.

es the density of the
gher the recycle, the
., more starch) in the
ike good gluten. The
)° Bé, contains 3–5%

ık is dewatered with
3 to 12% and then is
filtered to 40% solids
sold for animal feed
l 12% moisture (see
nd gluten-dewatering
teeping operation.

12

10

tem. 1 = feed; 2 = inlet
entripetal pump, medium
ate chamber; 9 = nozzles;
wash water. (Courtesy of

The equipment used to dewater heavy gluten (besides rotary, cloth-belt, and vacuum filters) are plate-type pressure filters (used where labor costs are low) and solid-bowl centrifuges. Lowering the SO_2 concentration to the steeps, heating the feed, and raising its pH from the normal level of 4.5 to as high as 7.0 increase the dewatering ability of the solid-bowl centrifuge.

STARCH PURIFICATION

Starch from the primary centrifuges contains 3–5% protein and small amounts of other soluble and insoluble impurities. The crude starch is washed with water in countercurrent fashion, using hydroclones that are 10 mm in diameter and grouped into clusters enclosed in housings capable of holding as many as 720 cyclones (Figs. 10 and 11). These units are then arranged into 10–14 separate stages operating in series. This is more than the six to eight stages required to wash potato starch (potato contains much less protein than does corn). The installations are compact and sanitary, have no moving parts (except the pumps that feed them), and are easy to automate. The efficiency of a hydroclone depends on pressure drop, control of the underflow orifice, feed solids concentration, and particle and fluid properties. About 2.1–2.5 kg of fresh water per kilogram of dry starch is used to extract the soluble impurities in the starch, and the hydroclone action mechanically separates the remaining insoluble gluten. The washed starch should contain <0.30% total protein and 0.01% soluble protein. These results are attainable with normal dent corn.

The water used for starch washing is generally deionized. It can be supplemented with a suitably pure condensate. The water is heated to 38–43°C (100–110°F) to enhance removal of soluble matter. The lower temperature is currently more prevalent because more stages of hydroclones are being used and more heat is generated from the additional pumps. Temperature sensors in the hydroclone systems protect the starch slurry from temperature exceeding 54°C (129°F), well below the starch-pasting temperature of 63°C (145°F). A heat exchanger is

Fig. 10. A starch-washing hydroclone. (Courtesy of the Center for Crops Utilization Research, Iowa State University)

STARCH:
Chemistry and Technology

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VOLUME I

FUNDAMENTAL ASPECTS

1965

Academic Press New York and London

Ch. 7
pp. 292

Evidence Appendix B-4:
Leach, Whistler et al., *Starch: Chemistry and Technology*, Table 1, page 292.

the gelatinization temperature is not affected by the presence of the linear starch fraction, and, hence, normal and waxy starches of the same species usually gelatinize over the same temperature range. Wrinkled pea and high-amylose corn starches show exceptional behavior because they are composed predominantly of linear molecules so highly associated that some of the granules even resist gelatinization in boiling water.

Table I

Gelatinization Characteristics of Native Starches

Starch			At 95°		
Species	*Type*	*Kofler gel. temp. range (°C) (9)*	*Swelling power[a]*	*Solubility (%)*	*Critical concentration value[a]*
Potato	Tuber	56–66	>1000	82	<0.1
Sago	Pith	—	97	39	1.0
Tapioca	Root	58.5–70	71	48	1.4
Canna	Root	—	72	37	1.4
Arrowroot	Root	—	54	28	1.9
Sweet potato	Root	—	46	18	2.2
Corn	Cereal	62–72	24	25	4.4
Sorghum	Cereal	68.5–75	22	22	4.8
Wheat	Cereal	52–63	21	41	5.0
Rice	Cereal	61–77.5	19	18	5.6
Waxy maize	Cereal	63–72	54	23	1.6
Waxy rice	Cereal	—	56	13	1.8
Waxy sorghum	Cereal	67.5–74	49	19	2.1
Wrinkled pea	Legume	—	6	19	20.0
High-amylose corn	Cereal	—	6	12	20.0
Chick pea (Garbanzo)	Legume	—	13	15	8.3

[a] See text. P 296 (7-9)

The many methods that have been proposed for measuring the gelati
zation temperature of starch have been discussed in detail by Kerr
Three different criteria have been used to detect the gelatinisation t
perature: loss of birefringence, increase in optical transmittancy, and ri
viscosity. Measurement of the loss of birefringence is the most sensi
accurate, and reproducible technique for determining the initial gelati
tion of starch. Methods based on the other two criteria lack sensitivity
Kofler microscope hot-stage provides a simple and rapid means for dete
the loss of birefringence (*9*, *16*). With this instrument, sigmoid gelatini
curves may be obtained by graphing the percentage of granules that

Carbohydrate Chemistry *for* Food Scientists

Roy L. Whistler and James N. BeMiller

eagan press
St. Paul, Minnesota, USA

Chart p 121

Evidence Appendix B-5:
Whistler et al., *Carbohydrate Chemistry for Food Scientists*, Table 6.1 on page 121, and page 128.

amylopectin in its starch, was so termed because, when the kernel is cut, the new surface appears vitreous or waxy, but there is no wax present. Other all-amylopectin starches are also called *waxy*.

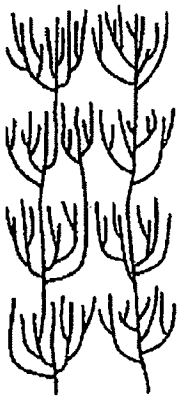
Potato amylopectin is unique in having phosphate ester groups attached to one in about every 200–550 α-D-glucopyranosyl units. The phosphate ester groups are located near branch points, most often (60–70%) at an O-6 position, but some also at O-3 positions. About 88% are found on B chains. Due to its phosphate ester content, potato starch has a negative charge and a pK_a of 3.7. The resulting slight coulombic repulsion may contribute to the rapid swelling of potato starch granules in warm water and to the high viscosity, good clarity, and low rate of retrogradation of potato starch pastes.

Phytoglycogen, which is similar to amylopectin but has a higher degree of branching (about 10% of all linkages), is present in sweet corn in amounts of up to 25% and is water soluble.

TABLE 6.1
General Properties of Some Starch Granules and Pastes

	Corn Starch	Waxy Maize Starch	High-Amylose Corn Starch	Potato Starch	Tapioca Starch	Wheat Starch
Granule size, μm	2–30	2–30	2–24	5–100	4–35	2–55
% Amylose	28	<2	50–70	21	17	28
Gelatinization/ pasting temp. (°C)[a]	62–80	63–72	66–170[b]	58–65	52–65	52–85
Relative viscosity	Medium	Medium high	Very low[b]	Very high	High	Low
Paste rheology (body)[c]	Short	Long	Short	Very long	Long	Short
Paste clarity	Opaque	Slightly cloudy	Opaque	Translucent	Translucent	Cloudy
Tendency to gel/ retrograde	High	Very low	Very high	Medium to low	Medium	High
Lipid, % DS[d]	0.8	0.2	<0.1	<0.1	<0.1	0.9
Protein, % DS[d]	0.35	0.25	0.5	0.1	0.1	0.4
Phosphorus, % DS[d]	0.00	0.00	0.00	0.08	0.00	0.00

[a] From the initial temperature of gelatinization to complete cookout.
[b] Under ordinary cooking conditions, where the slurry is heated to 95–100°C, high-amylose corn starch produces essentially no viscosity. Cookout does not occur until the temperature reaches 160–170°C (320–340°F). However, loss of birefringence begins at about 66°C.
[c] See Chapter 5, footnote 1.
[d] DS = dry solids.



ortion of an amylopectin clusters (right). Individual ellical.

is about 10% for normal corn starch and about 25% for waxy maize starch. Reversible swelling generally increases with granule diameter, approaching a doubling in size for the largest potato starch granules. The equilibrium moisture content of corn starch granules in water is about 28%, and of potato starch granules about 33%.

Gelatinization refers to the disruption of molecular order within starch granules as they are heated in the presence of water. Evidence for the loss of organized structure includes irreversible granule swelling, loss of birefringence (Fig. 6.5), and loss of crystallinity. Leaching of amylose occurs during gelatinization, but some leaching of amylose occurs at temperatures below the gelatinization temperature due to its location in noncrystalline regions and the fact that it is a relatively small, linear molecule that can diffuse out of granules. Gelatinization occurs over a temperature range, with larger granules generally gelatinizing first and smaller granules later. The apparent temperature of initial gelatinization and the range over which gelatinization occurs (see later) depend on the method of measurement and the starch-water ratio, granule type, and heterogeneities within the granule population. Values obtained using a polarizing microscope equipped with a hot stage are the initiation temperature (when the first granule in the field loses birefringence), the midpoint temperature, and the completion or birefringence endpoint temperature (when the last granule in the field loses birefringence). Other methods for determining the temperature and/or heat of gelatinization involve measuring the absorption of heat energy, loss of turbidity, molecule dissolution, dye adsorption, rate of enzyme-catalyzed hydrolysis, and chemical reactivity and determining changes in the x-ray pattern. Of these methods, one of the most sensitive, and one easy to measure, is the increase in extent of hydrolysis catalyzed by glucoamylase, or a mixture of α-amylase and glucoamylase, with the resulting D-glucose determined quantitatively using glucose oxidase (see section on Enzyme-Catalyzed Hydrolysis). Gelatinization temperature data for several commercial starches are given in Table 6.1.

Continued heating of starch granules in excess water results in further granule swelling and additional leaching of soluble components (primarily amylose). If shear is applied at this stage, granules are disrupted and a paste is formed. A starch paste is a viscous mass consisting of a continuous phase (a molecular dispersion) of solubilized amylose and/or amylopectin and a discontinuous

RELATED PROCEEDINGS APPENDIX

NONE

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